Guide to Manually Refill the Liquid Nitrogen Tank of the LFA 447 Apparatus

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by

Hicham Johra

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1. Foreword

The aim of this lecture note is to explain how to safely manually fill up or refill the liquid nitrogen tank for the infra-red sensor of the Laser Flash Apparatus LFA 447 (Netzsch Gerätebau GmbH [1]) at the Building Material Characterization Laboratory of Aalborg University - Department of Civil Engineering [2].

Warnings: Liquid nitrogen is very cold: **-196 °C**. If getting in contact with clothes, skin or eyes, it can cause severe frostbite and cold burns. When pouring liquid nitrogen from a container to another, droplets of liquid nitrogen might be ejected up in the air. Protect yourself with adequate safety equipment, especially for your hands and your eyes.

2. Introduction

In order to conduct properly Laser Flash Analysis measurements with the LFA 447 apparatus, the infra-red sensor of the latter has to be cooled down with liquid nitrogen. Once filled up completely, and in the absence of automated refilling device, the liquid nitrogen reservoir of the LFA 447 apparatus (see *Figure 1*) enables to conduct Laser Flash Analysis measurements during approximately 4 hours. After that, all the liquid nitrogen in the reservoir has been evaporated and the infra-red sensor is not cold enough to insure proper measurements anymore.

Figure 1: *The Laser Flash FLA 447 apparatus (left), the location of the infra-red sensor module on the LFA 447 apparatus (center), removing the top cover (right).*

Once the liquid nitrogen reservoir is empty and the infra-red sensor is not cooled down anymore, the measurement signal becomes random and totally nonsensical from a Laser Flash Analysis standpoint (see *Figure 2*).

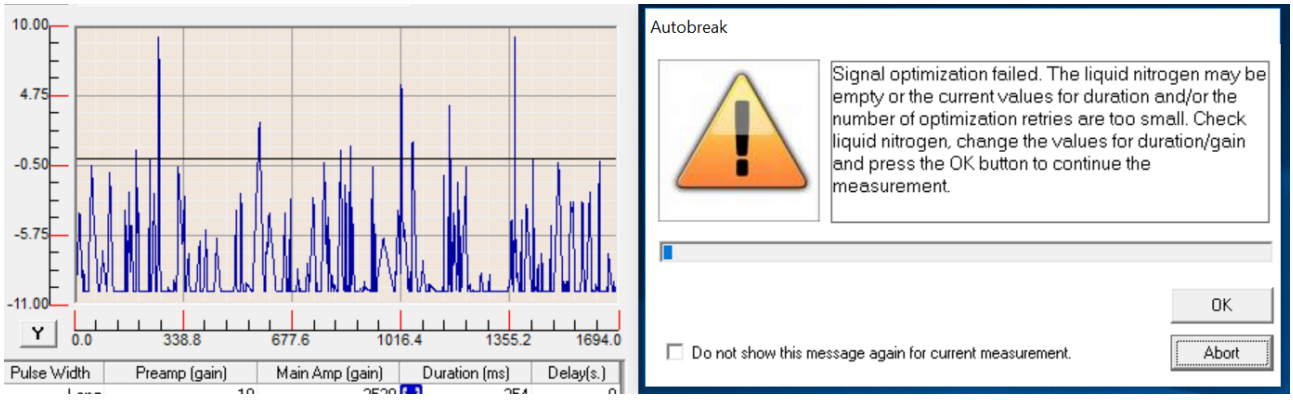

Figure 2: *Wrong measurement resulting from infra-red detector being too warm: nonsensical measurement signal (left); error message displayed by the control software.*

At this point, no correct measurement can be extracted from that random signal. If the LFA 447 is running measurements with the “Automatic Adjust” mode activated (see *Figure 3*), the apparatus will detect this wrong signal and will stop the measurement run and display an error message asking to refill the liquid

nitrogen tank to cool down the infra-red sensor again (see *Figure 2*). Refill the liquid nitrogen tank before resuming the current measurement run. If the liquid nitrogen tank is not refilled and the operator did not press the “OK” button on the error message window (see *Figure 2*), the software will automatically abort the current measurement after few minutes of waiting time.

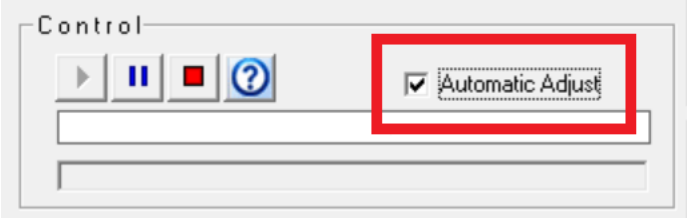

Figure 3: *LFA 447 running measurements with the ”Automatic Adjust” mode activated.*

3. Safety first !

When handling liquid nitrogen or equipment in direct contact with liquid nitrogen, make sure that you wear all the necessary safety and protection equipment (see *Figure 4*):

- Protection goggles
- Cryogenic protection gloves
- Cryogenic protection apron
- Safety shoes

It is recommended that other people in the room where liquid nitrogen is being handled wear protection goggles, even if they do not manipulate the liquid nitrogen themselves. Protection goggles are very important to avoid eye injuries in case of spray or splash of liquid nitrogen droplets.

Figure 4: *Safety and protection equipment for the handling of liquid nitrogen.*

One can find the safety and protection equipment for the handling of liquid nitrogen inside the dedicated (see *Figure 5*).

Figure 5: *Location of the safety and protection equipment in the dedicated compartment drawer under the Laser Flash apparatus LFA 447.*

4. Fill up the liquid nitrogen transfer vessel

To safely transfer liquid nitrogen from the storage dewar located in the laboratory nearby the Laser Flash apparatus (see *Figure 6*) to the liquid nitrogen tank of the Laser Flash apparatus (see *Figure 1*), use of the liquid nitrogen transfer vessel (see *Figure 7*). You can find the latter in the drawer of the safety and protection equipment under the Laser Flash apparatus LFA 447 (see *Figure 5*).

Figure 6: *Liquid nitrogen storage dewar located nearby the Laser Flash apparatus.*

Figure 7: *Liquid nitrogen transfer vessel.*

Make sure that the liquid nitrogen storage dewar is pressurized at around 0.5 bar (see *Figure 8*). If this is not the case and the pressure inside the dewar is lower than 0.4 bar, the latte is empty and must be refilled before use. Contact the technical staff to refill the liquid nitrogen storage dewar or follow the dedicated lecture note explaining how to refill the liquid nitrogen storage dewar.

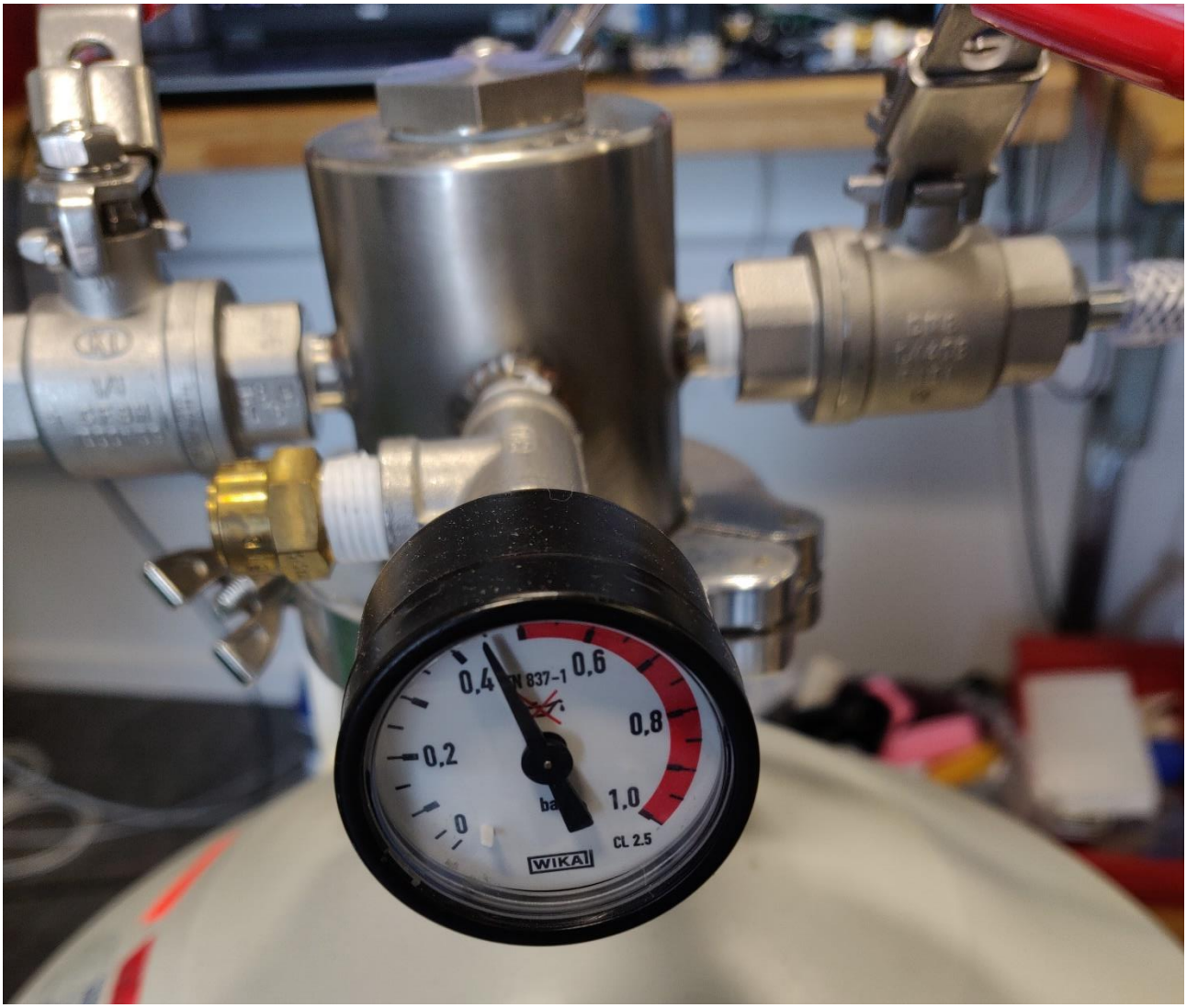

Figure 8: *Adequate pressure for the liquid nitrogen storage dewar.*

Use the valve connected to a plastic tube to transfer liquid nitrogen into the transfer vessel. Hold the liquid nitrogen transfer vessel next to the storage dewar with all necessary safety and protection equipment (especially the cryogenic protection gloves and the protection goggles). Place the plastic tube inside the liquid nitrogen transfer vessel and open carefully the valve (see *Figure 9*). The liquid nitrogen is pressurized and the plastic tube can rapidly move away from the transfer vessel if the valve is opened to quickly. Fill up the transfer vessel carefully and mind the liquid nitrogen splash.

Figure 9: *Using the valve connected to the plastic tube to transfer liquid nitrogen into the transfer vessel.*

5. Refill the liquid nitrogen tank of the LFA 447

To refill the liquid nitrogen tank of the LFA 447, remove the top cover from the infra-red sensor module on the apparatus (see *Figure 10*).

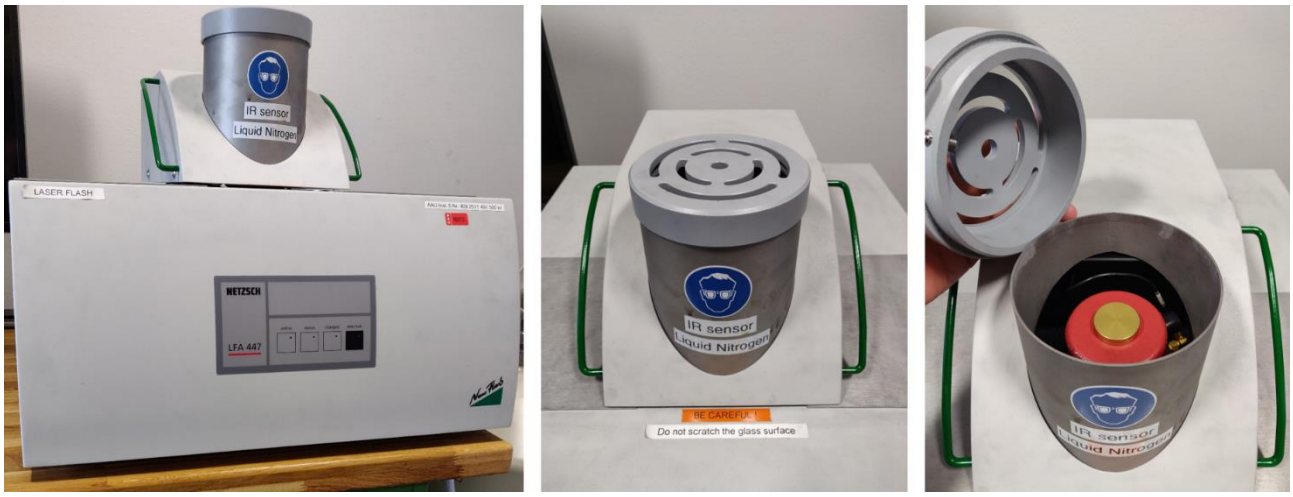

Figure 10: *The Laser Flash FLA 447 apparatus (left), the location of the infra-red sensor module on the LFA 447 apparatus (center), removing the top cover (right).*

Remove the plug from the top of the infra-red sensor module and insert the funnel into the free opening of the sensor's liquid nitrogen tank (see *Figure 11*).

Figure 11: *Removing the top cover (left), removing the plug from the liquid nitrogen tank (center), inserting the funnel into the free opening of the tank (right).*

While still wearing all the necessary safety and protection equipment (especially the cryogenic protection gloves and the protection goggles), pour carefully the liquid nitrogen into the funnel (see *Figure 12*). If the infra-red sensor's tank has been empty for a long time, the inside of the tank is "warm" and therefore the first liquid nitrogen pouring will be vaporized immediately (see *Figure 12*). In that case, after a first pouring into the funnel, remove the funnel from the free opining of the tank and wait until no more vapor escapes

from it. Then put the funnel back in place and pour approximately 200 ml of liquid nitrogen in several smaller pourings. The entire content of the liquid nitrogen transfer vessel should be sufficient to entirely refill the tank of the apparatus. If continuing pouring liquid nitrogen into the funnel while the tank is full, some liquid nitrogen will escape directly from the free opening of the apparatus tank and flow down the sides of the apparatus (see *Figure 12*). This is the indication that the apparatus tank is full and that the operator should stop pouring more liquid nitrogen into the funnel. Wait a couple of minutes so that the temperature of the infra-red detector and the liquid nitrogen tank stabilizes.

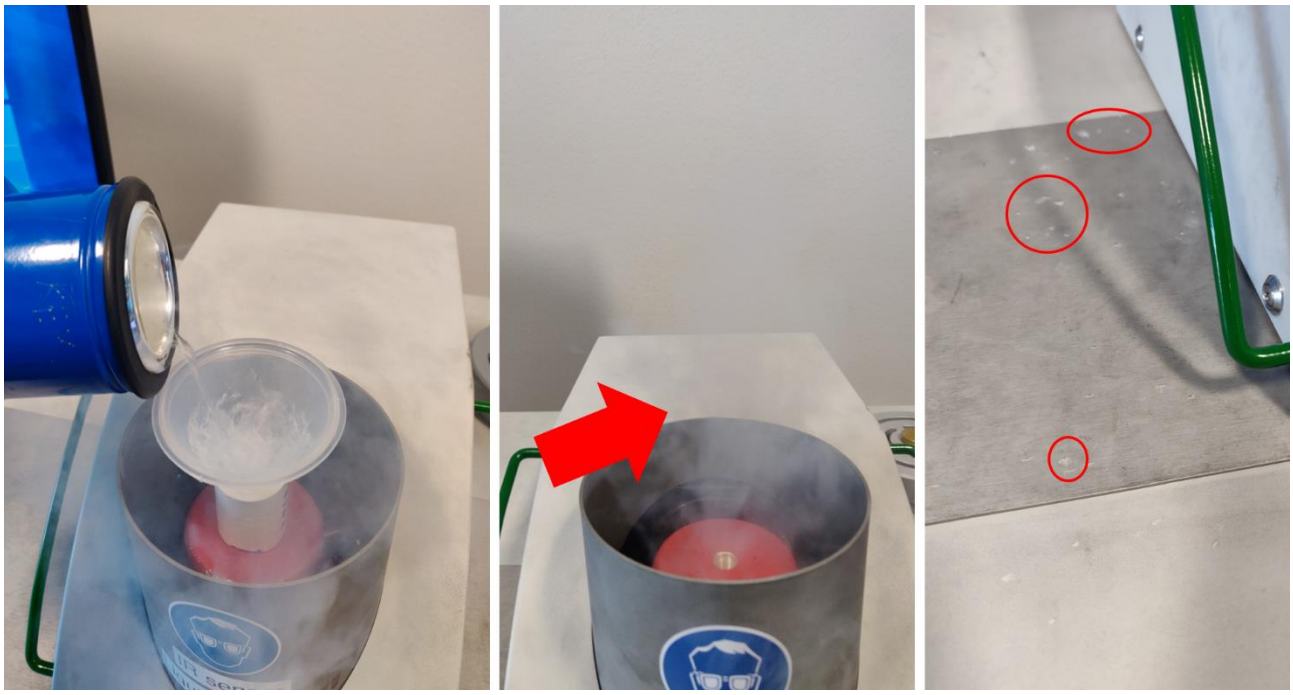

Figure 12: *Pouring liquid nitrogen into the funnel (left); a cloud will form above the liquid nitrogen tank if the infra-red sensor is "warm" and evaporates a lot of liquid nitrogen after the first pouring (center); liquid nitrogen droplets flowing down the sides of the apparatus indicating that the liquid nitrogen tank is full (right).*

Once the refill operation is finished, DO NOT FORGET to remove the funnel from the opening of the tank, put back the plug on the apparatus tank, and place back the top cover of the infra-red sensor module.

The Laser Flash apparatus is now ready to use. If the measurement parameters had been inputted already and the measurement session had been interrupted while running (see *Figure 2*), resuming the measurement procedure by pressing the "OK" button of the error message if it is still visible (see *Figure 2*), or press the green arrow "start" button to start a new run of measurements (see *Figure 13*).

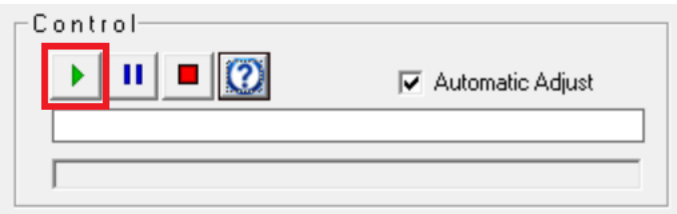

Figure 13: *Start a new run of measurements.*

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[1] Netzsch Gerätebau GmbH. Operating Instructions Nano-Flash-Apparatus LFA 447, 2001.

[2] Building Material Characterization Laboratory of Aalborg University, Department of Civil Engineering, Aalborg, Denmark.
https://buildingmaterials.civil.aau.dk

[3] Hicham Johra. Description of the laser flash analysis method for thermal diffusivity measurement with the LFA 447. DCE Lecture Notes No. 73. Department of Civil Engineering, Aalborg University, 2019.
https://vbn.aau.dk/ws/portalfiles/portal/312969074/Description_of_the_Laser_Flash_Analysis_Method_for_Thermal_Diffusivity_Measurement_with_the_LFA_447.pdf

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